

4. Digestions

4.3 Boiling Digestions

4.3.3 Digestion with HClO₄: fish meat and fish bones

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All works take place under the fume hood! Check if the available fume hood is suitable for HClO₄!

Day 1: Preparation and HClO₄ digestion

- ▶ Put on protective clothing (gloves, apron, glasses).
- ▶ Weigh in 250 mg dried fish sample in Teflon vessels (Heinrichs et al. 1986).
- ▶ To oxidize the organic substance 3 ml conc. HNO₃ are added and the samples are heated to 60 °C on a hotplate for 1 hour.
- ▶ Subsequently, 3 ml conc. HClO₄ are added and the Teflon vessels are closed.
- ▶ The vessels are heated in the oven to 185 °C for 12 hours.

Day 2: Quantitative transfer for the determination of elements

- ▶ Afterwards, the vessels are opened carefully and
- ▶ the acids are evaporated at 185 °C on the heating plate until the samples are almost dry.
- ▶ Subsequently, the samples are evaporated 3 times with 2 ml 1 + 1 HCl.
- ▶ Add 5 ml 2 vol. % HNO₃ to the samples and handle for 1 h at 60 °C.
- ▶ After cooling down, the sample is transferred into a 50 ml centrifuge tube and
- ▶ rinsed with 2 vol. % HNO₃ from the Teflon vessel and filled with 2 vol. % HNO₃ up to 50 ml in the centrifuge tube.
- ▶ The determination of element took place at ICP-OES at IOW (iCAP 7400 Duo, Thermo Fisher Scientific).

Reference

Heinrichs, H, Brumsack, HJ, Loftfield, N, König, N (1986) Verbessertes Druckaufschlußsystem für biologische und anorganische Materialien. J Plant Nutr Soil Sci 149, 350-353, DOI: [10.1002/jpln.19861490313](https://doi.org/10.1002/jpln.19861490313)

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